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(56) Documents Cited

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(58) Field of Search

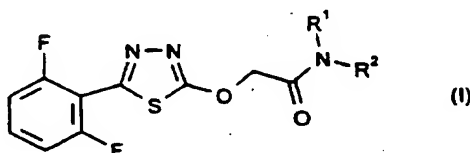
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(54) N-substituted 2-[2-(2,6-difluoro-phenyl)-1,3,4-thiadiazol-5-yl-oxy]-acetamides as herbicides

(57) The invention relate to new 2-[2-(2,6-difluoro-phenyl)-1,3,4-thiadiazol-5-yl-oxy]-acetamides of the formula (I)



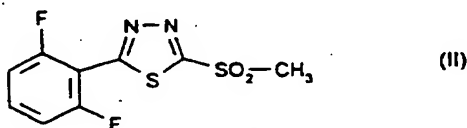
in which

R¹ represents alkyl, alkenyl, alkynyl or arylalkyl, each of which is optionally substituted, and

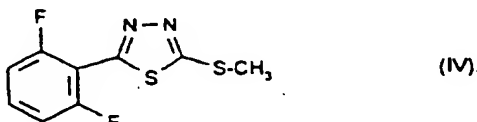
R² represents alkyl, alkenyl, alkynyl, cycloalkyl, cycloalkenyl, alkoxy, alkenyloxy, alkynyloxy, arylalkyl or aryl, each of which is optionally substituted or

R¹ and R², together with the nitrogen atom to which they are bonded, represent an optionally substituted monocyclic or bicyclic heterocycle;

a process and new intermediate of formulae (II) and (IV) for their preparation; and their use as herbicides.



and



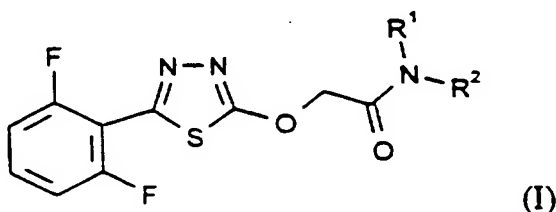
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2-[2-(2,6-Difluoro-phenyl)-1,3,4-thiadiazol-5-yl-oxy]-acetamides

The invention relates to new 2-[2-(2,6-difluoro-phenyl)-1,3,4-thiadiazol-5-yl-oxy]-acetamides, a process and new intermediates for their preparation and their use as herbicides.

10 It has been disclosed that certain 2-[2-(fluorophenyl)-1,3,4-thiadiazol-5-yl-oxy]-acetamides, such as e.g. the compound N-methyl-N-phenyl-2-[2-(3-fluoro-phenyl)-1,3,4-thiadiazol-5-yl-oxy]-acetamide, have herbicidal properties (cf. EP-A 537 543). The herbicidal action of these compounds, however, in particular at low application rates or active compound concentrations, is not always entirely satisfactory.

15 The new 2-[2-(2,6-difluoro-phenyl)-1,3,4-thiadiazol-5-yl-oxy]-acetamides of the general formula (I) have now been found



in which

R¹ represents alkyl, alkenyl, alkynyl or arylalkyl, each of which is optionally

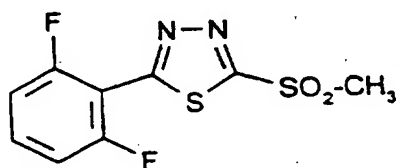
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substituted. and

R^2 represents alkyl, alkenyl, alkynyl, cycloalkyl, cycloalkenyl, alkoxy, alkenyloxy, alkynyloxy, arylalkyl or aryl, each of which is optionally substituted, or

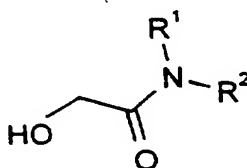
5 R^1 and R^2 , together with the nitrogen atom to which they are bonded, represent an optionally substituted, monocyclic or bicyclic heterocycle.

It has further been found that the new 2-[2-(2,6-difluoro-phenyl)-1,3,4-thiadiazol-5-yl-oxy]-acetamides of the general formula (I) are obtained when 2-(2,6-difluoro-phenyl)-5-methylsulphonyl-1,3,4-thiadiazole of the formula (II)



(II)

10 is reacted with hydroxyacetamides of the general formula (III)



(III)

in which

R^1 and R^2 have the meaning indicated above,

15 if appropriate in the presence of a diluent and if appropriate in the presence of an acid-binding agent.

Finally, it has been found that the new 2-[2-(2,6-difluoro-phenyl)-1,3,4-thiadiazol-5-yl-

oxy]-acetamides of the general formula (I) have interesting herbicidal properties.

Surprisingly, the new compounds of the formula (I) show, combined with good tolerability to crop plants, such as e.g. barley, substantially stronger herbicidal action than the known, structurally similar compound N-methyl-N-phenyl-2-[2-(3-fluorophenyl)-1,3,4-thiadiazol-5-yl-oxy]-acetamide.

The invention preferably relates to compounds of the formula (I), in which

R¹ represents C₁-C₈-alkyl (which is optionally substituted by fluorine, chlorine, cyano or C₁-C₄-alkoxy), C₂-C₈-alkenyl (which is optionally substituted by fluorine and/or chlorine), C₂-C₈-alkinyl or benzyl (which is optionally substituted by fluorine, chlorine, cyano, C₁-C₄-alkyl or C₁-C₄-alkoxy), and

R² represents C₁-C₈-alkyl (which is optionally substituted by fluorine, chlorine, cyano or C₁-C₄-alkoxy), C₂-C₈-alkenyl (which is optionally substituted by fluorine and/or chlorine), C₂-C₈-alkinyl, C₃-C₆-cycloalkyl (which is optionally substituted by chlorine and/or C₁-C₃-alkyl), cyclopentenyl or cyclohexenyl, C₁-C₈-alkoxy (which is optionally substituted by methoxy or ethoxy), C₃-C₄-alkenyloxy, benzyl (which is optionally substituted by fluorine, chlorine, cyano, C₁-C₄-alkyl or C₁-C₄-alkoxy) or phenyl (which is optionally substituted by fluorine, chlorine, bromine, cyano, nitro, C₁-C₄-alkyl, trifluoromethyl, C₁-C₄-alkoxy or C₁-C₄-alkylthio), or

R¹ and R², together with the nitrogen atom to which they are bonded, represent a monocyclic or bicyclic, five- to seven-membered nitrogen heterocycle which is optionally mono- to trisubstituted by C₁-C₃-alkyl.

The invention relates in particular to compounds of the formula (I), in which

R¹ represents methyl, ethyl, n- or i-propyl, n-, i-, s- or t-butyl, n-, i-, s- or t-pentyl (which in each case are optionally substituted by fluorine, chlorine, cyano, methoxy or ethoxy), propenyl, butenyl, pentenyl, propinyl, butinyl, pentinyl or

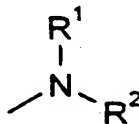
benzyl,

R² represents methyl, ethyl, n- or i-propyl, n-, i- or s-butyl, n-, i- or s-pentyl, n-, i- or s-hexyl (which in each case are optionally substituted by fluorine, chlorine, cyano, methoxy or ethoxy), propenyl, butenyl, pentenyl, propinyl, butinyl or pentinyl, cyclopentyl or cyclohexyl (which in each case are optionally substituted by methyl and/or ethyl), cyclohexenyl, methoxy, ethoxy, n- or i-propoxy, n-, i- or s-butoxy, n-, i- or s-pentyloxy (which in each case are optionally substituted by methoxy or ethoxy), benzyl (which is optionally substituted by fluorine, chlorine and/or methyl) or phenyl (which is optionally substituted by fluorine, chlorine, bromine, cyano, nitro, methyl, ethyl, trifluoromethyl, methoxy or ethoxy), or

R¹ and R², together with the nitrogen atom to which they are bonded, represent piperidinyl which is optionally mono- to trisubstituted by methyl and/or ethyl, pyrrolidinyl which is optionally mono- or disubstituted by methyl and/or ethyl, perhydroazepinyl or 1,2,3,4-tetrahydro(iso)quinolinyl.

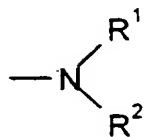
The abovementioned definitions of radicals indicated generally or in preferred ranges apply both to the final products of the formula (I) and correspondingly to the starting substances or intermediates in each case needed for their preparation. These definitions of radicals can be combined with one another in any desired manner, i.e. also between the indicated ranges of preferred compounds.

Examples of the possible meanings of the group



in the formula (I) are shown in Table 1 below.

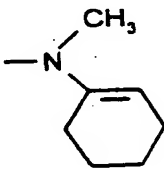
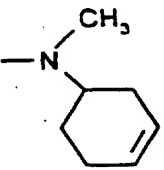
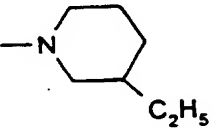
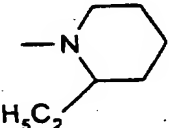
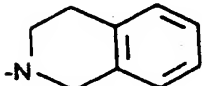

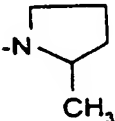
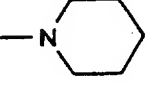
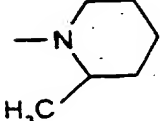
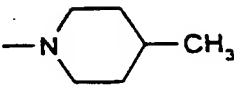
Table 1: Examples of the meaning of the group



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$\begin{array}{c} \text{R}^1 \\ \diagup \\ -\text{N} \\ \diagdown \\ \text{R}^2 \end{array}$	$\begin{array}{c} \text{R}^1 \\ \diagup \\ -\text{N} \\ \diagdown \\ \text{R}^2 \end{array}$
$-\text{N}(\text{CH}_3)_2$	$-\text{N}(\text{C}_4\text{H}_9)_2$
$-\text{N}(\text{C}_2\text{H}_5)_2$	$-\text{N}(\text{CH}_2\text{CH}=\text{CH}_2)_2$
$-\text{N}(\text{C}_3\text{H}_7)_2$	$-\text{N}(\text{CH}_2\text{C}\equiv\text{CH})_2$
$\begin{array}{c} \text{CH}_3 \\ \diagup \\ -\text{N} \\ \diagdown \\ \text{CH}_2\text{CF}_3 \end{array}$	$\begin{array}{c} \text{CH}_3 \\ \diagup \\ -\text{N} \\ \diagdown \\ \text{CH}_2\text{C}\equiv\text{CH} \end{array}$
$\begin{array}{c} \text{CH}(\text{CH}_3)_2 \\ \diagup \\ -\text{N} \\ \diagdown \\ \text{OCH}_2\text{CH}_2\text{OC}_2\text{H}_5 \end{array}$	$\begin{array}{c} \text{CH}_3 \\ \diagup \\ -\text{N} \\ \diagdown \\ \text{CHCH}_2\text{C}_2\text{H}_5 \\ \\ \text{CH}_3 \end{array}$
$\begin{array}{c} \text{CH}_3 \\ \diagup \\ -\text{N} \\ \diagdown \\ \text{CH}_2\text{OCH}_3 \end{array}$	$\begin{array}{c} \text{CH}_3 \\ \diagup \\ -\text{N} \\ \diagdown \\ \text{C}_6\text{H}_{11} \end{array}$

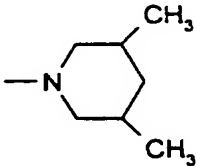
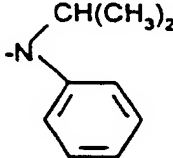
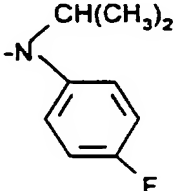
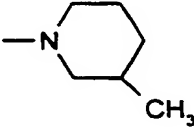
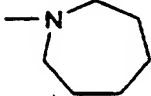
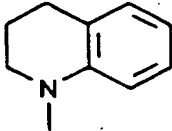
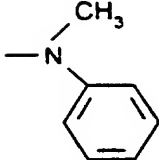
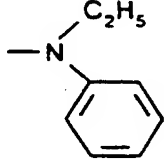
Table 1 (Continued)

$\text{---N} \begin{matrix} \text{R}^1 \\ \text{R}^2 \end{matrix}$	$\text{---N} \begin{matrix} \text{R}^1 \\ \text{R}^2 \end{matrix}$
	
	
	
	
	

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Table 1

(Continued)

$\begin{array}{c} R^1 \\ \\ -N \\ \\ R^2 \end{array}$	$\begin{array}{c} R^1 \\ \\ -N \\ \\ R^2 \end{array}$
	
	
	
	

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Table 1

(Continued)

$\begin{array}{c} R^1 \\ \\ -N \\ \\ R^2 \end{array}$	$\begin{array}{c} R^1 \\ \\ -N \\ \\ R^2 \end{array}$
$\begin{array}{c} CH(CH_3)_2 \\ \\ -N \\ \\ F \end{array}$	$\begin{array}{c} CH(CH_3)_2 \\ \\ -N \\ \\ F \end{array}$
$\begin{array}{c} CH(CH_3)_2 \\ \\ -N \\ \\ Cl \end{array}$	$\begin{array}{c} CH_3 \\ \\ -N \\ \\ H_3C \end{array}$
$\begin{array}{c} CH(CH_3)_2 \\ \\ -N \\ \\ CH_3 \end{array}$	$\begin{array}{c} CH(CH_3)_2 \\ \\ -N \\ \\ OCH_3 \end{array}$
$\begin{array}{c} CH(CH_3)_2 \\ \\ -N \\ \\ CH_3 \end{array}$	$\begin{array}{c} CH(CH_3)_2 \\ \\ -N \\ \\ CF_3 \end{array}$

5

Table 1

(Continued)

	$\begin{array}{c} \text{R}^1 \\ \diagup \\ \text{---N} \\ \diagdown \\ \text{R}^2 \end{array}$	$\begin{array}{c} \text{R}^1 \\ \diagup \\ \text{---N} \\ \diagdown \\ \text{R}^2 \end{array}$
	$\begin{array}{c} \text{CH}_3 \\ \diagup \\ \text{---N} \\ \diagdown \\ \text{C}_2\text{H}_5 \end{array}$	$\begin{array}{c} \text{CH}_3 \\ \diagup \\ \text{---N} \\ \diagdown \\ \text{C}_3\text{H}_7 \end{array}$
	$\begin{array}{c} \text{CH}_3 \\ \diagup \\ \text{---N} \\ \diagdown \\ \text{CH}(\text{CH}_3)_2 \end{array}$	$\begin{array}{c} \text{CH}_3 \\ \diagup \\ \text{---N} \\ \diagdown \\ \text{C}_4\text{H}_9 \end{array}$
5	$\begin{array}{c} \text{CH}_3 \\ \diagup \\ \text{---N} \\ \diagdown \\ \text{CH}_2\text{CH}(\text{CH}_3)_2 \end{array}$	$\begin{array}{c} \text{C}_3\text{H}_7 \\ \diagup \\ \text{---N} \\ \diagdown \\ \text{CHC}_2\text{H}_5 \\ \\ \text{CH}_3 \end{array}$
	$\begin{array}{c} \text{C}_2\text{H}_5 \\ \diagup \\ \text{---N} \\ \diagdown \\ \text{C}_3\text{H}_7 \end{array}$	$\begin{array}{c} \text{C}_2\text{H}_5 \\ \diagup \\ \text{---N} \\ \diagdown \\ \text{CH}(\text{CH}_3)_2 \end{array}$
	$\begin{array}{c} \text{C}_2\text{H}_5 \\ \diagup \\ \text{---N} \\ \diagdown \\ \text{C}_4\text{H}_9 \end{array}$	$\begin{array}{c} \text{C}_2\text{H}_5 \\ \diagup \\ \text{---N} \\ \diagdown \\ \text{CH}_2\text{CH}(\text{CH}_3)_2 \end{array}$
	$\begin{array}{c} \text{C}_2\text{H}_5 \\ \diagup \\ \text{---N} \\ \diagdown \\ \text{CHC}_2\text{H}_5 \\ \\ \text{CH}_3 \end{array}$	$\begin{array}{c} \text{C}_3\text{H}_7 \\ \diagup \\ \text{---N} \\ \diagdown \\ \text{CH}(\text{CH}_3)_2 \end{array}$

Table 1

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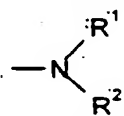
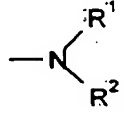
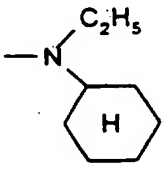
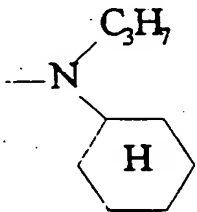
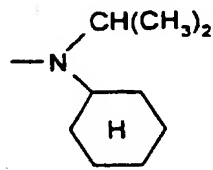
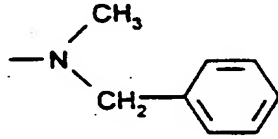
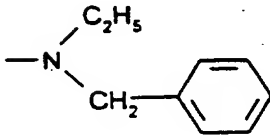
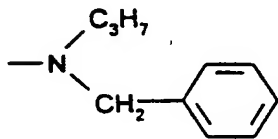
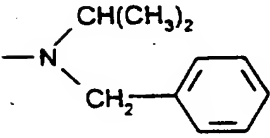
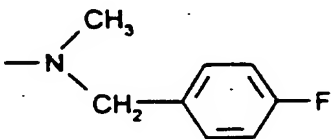
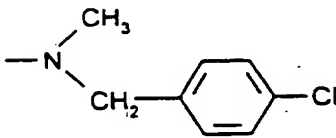
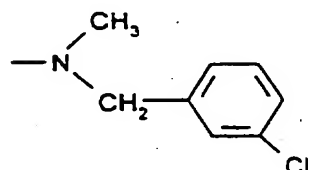
		
		
		
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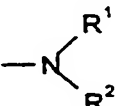
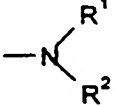
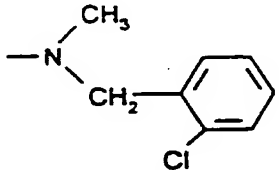
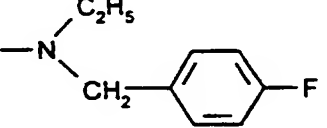
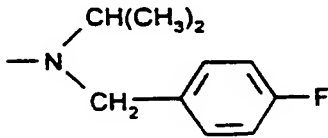
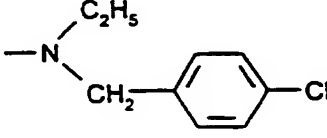
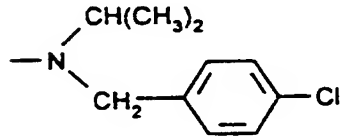
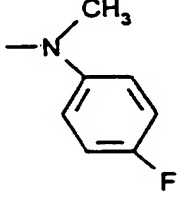
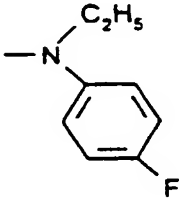
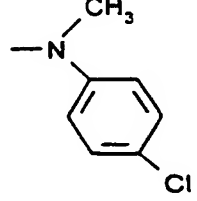
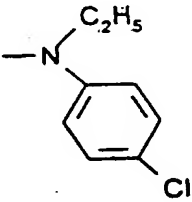
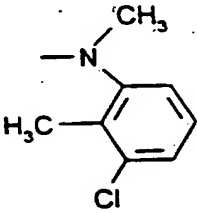
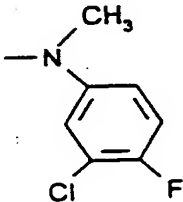
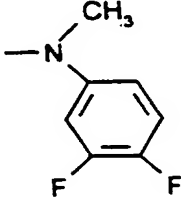
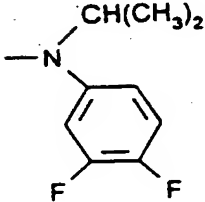
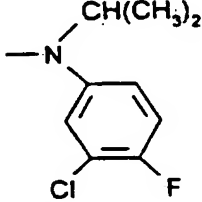
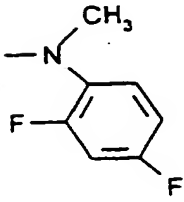
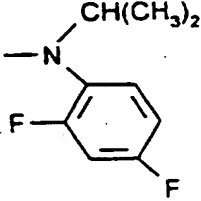
		
5		
		
		
		

Table 1

(Continued)

$\text{—N—}\begin{matrix} \text{R}^1 \\ \text{R}^2 \end{matrix}$	$\text{—N—}\begin{matrix} \text{R}^1 \\ \text{R}^2 \end{matrix}$
	
	
	
	

5

Table 1 (Continued)

$\text{—N} \begin{matrix} \text{R}^1 \\ \text{R}^2 \end{matrix}$	$\text{—N} \begin{matrix} \text{R}^1 \\ \text{R}^2 \end{matrix}$
<chem>CN(C)c1cc(C)c(C)cc1</chem>	<chem>CC(C)Nc1ccc(C(F)(F)F)cc1</chem>
<chem>CN(C)c1ccc(C(F)(F)F)cc1</chem>	<chem>CN(C)c1cc(C(F)(F)F)cc(C(F)(F)F)c1</chem>
<chem>CN(C)c1cc(C)cc(C)cc1</chem>	<chem>CC(C)Nc1cc(F)cc(F)cc1</chem>

5

Table 1

(Continued)

$\begin{array}{c} \text{R}^1 \\ \diagup \\ \text{---N} \\ \diagdown \\ \text{R}^2 \end{array}$	$\begin{array}{c} \text{R}^1 \\ \diagup \\ \text{---N} \\ \diagdown \\ \text{R}^2 \end{array}$
$\begin{array}{c} \text{CH(CH}_3)_2 \\ \\ \text{---N} \\ \\ \text{C}_6\text{H}_3\text{Cl}_2 \end{array}$	$\begin{array}{c} \text{CH(CH}_3)_2 \\ \\ \text{---N} \\ \\ \text{C}_6\text{H}_3\text{CH}_3 \end{array}$
$\begin{array}{c} \text{CH}_3 \\ \\ \text{---N} \\ \\ \text{C}_6\text{H}_3\text{Cl}_2 \end{array}$	$\begin{array}{c} \text{CH}_3 \\ \\ \text{---N} \\ \\ \text{CH}_2\text{CH}_2\text{CN} \end{array}$
$\begin{array}{c} \text{C}_2\text{H}_5 \\ \\ \text{---N} \\ \\ \text{CH}_2\text{CH}_2\text{CN} \end{array}$	$\text{---N(CH}_2\text{CH}_2\text{CN)}_2$
$\begin{array}{c} \text{CH(CH}_3)_2 \\ \\ \text{---N} \\ \\ \text{CH}_2\text{CH}_2\text{OCH}_3 \end{array}$	$\begin{array}{c} \text{C}_2\text{H}_5 \\ \\ \text{---N} \\ \\ \text{CH}_2\text{CH}_2\text{OCH}_3 \end{array}$
$\begin{array}{c} \text{CH}_3 \\ \\ \text{---N} \\ \\ \text{CH}_2\text{CH}_2\text{OCH}_3 \end{array}$	$\begin{array}{c} \text{CH}_3 \\ \\ \text{---N} \\ \\ \text{OC}_2\text{H}_5 \end{array}$

5

Table 1

(Continued)

$\begin{array}{c} \text{R}^1 \\ \diagup \\ \text{---N} \\ \diagdown \\ \text{R}^2 \end{array}$	$\begin{array}{c} \text{R}^1 \\ \diagup \\ \text{---N} \\ \diagdown \\ \text{R}^2 \end{array}$
$\begin{array}{c} \text{CH}_3 \\ \diagup \\ \text{---N} \\ \diagdown \\ \text{OC}_3\text{H}_7 \end{array}$	$\begin{array}{c} \text{CH}_3 \\ \diagup \\ \text{---N} \\ \diagdown \\ \text{OC}_4\text{H}_9 \end{array}$
$\begin{array}{c} \text{C}_2\text{H}_5 \\ \diagup \\ \text{---N} \\ \diagdown \\ \text{OC}_2\text{H}_5 \end{array}$	$\begin{array}{c} \text{C}_2\text{H}_5 \\ \diagup \\ \text{---N} \\ \diagdown \\ \text{OC}_3\text{H}_7 \end{array}$
$\begin{array}{c} \text{C}_2\text{H}_5 \\ \diagup \\ \text{---N} \\ \diagdown \\ \text{OC}_4\text{H}_9 \end{array}$	$\begin{array}{c} \text{C}_3\text{H}_7 \\ \diagup \\ \text{---N} \\ \diagdown \\ \text{OC}_3\text{H}_7 \end{array}$
$\begin{array}{c} \text{C}_3\text{H}_7 \\ \diagup \\ \text{---N} \\ \diagdown \\ \text{OC}_4\text{H}_9 \end{array}$	$\begin{array}{c} \text{CH}(\text{CH}_3)_2 \\ \diagup \\ \text{---N} \\ \diagdown \\ \text{OC}_2\text{H}_5 \end{array}$
$\begin{array}{c} \text{CH}(\text{CH}_3)_2 \\ \diagup \\ \text{---N} \\ \diagdown \\ \text{OC}_3\text{H}_7 \end{array}$	$\begin{array}{c} \text{CH}(\text{CH}_3)_2 \\ \diagup \\ \text{---N} \\ \diagdown \\ \text{OCH}_2\text{CH}_2\text{OCH}_3 \end{array}$
$\begin{array}{c} \text{CH}_3 \\ \diagup \\ \text{---N} \\ \diagdown \\ \text{C}_6\text{H}_4\text{Cl} \end{array}$	$\begin{array}{c} \text{CH}_3 \\ \diagup \\ \text{---N} \\ \diagdown \\ \text{C}_6\text{H}_4\text{CH}_3 \end{array}$

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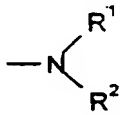
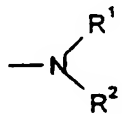
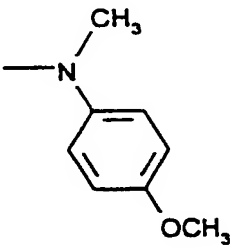
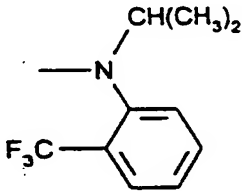
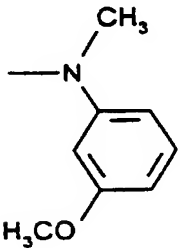
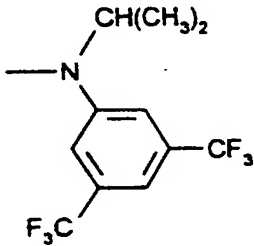
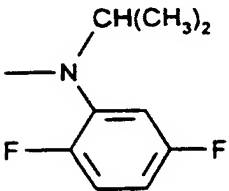
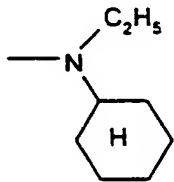
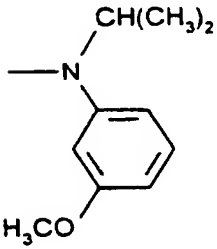
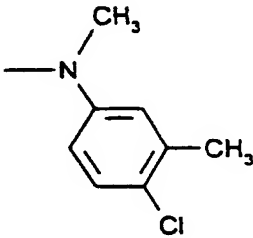
Table 1

(Continued)

$\begin{array}{c} R^1 \\ \\ -N- \\ \\ R^2 \end{array}$	$\begin{array}{c} R^1 \\ \\ -N- \\ \\ R^2 \end{array}$
$\begin{array}{c} CH(CH_3)_2 \\ \\ -N- \\ \\ \text{C}_6\text{H}_4 \\ \\ Cl \end{array}$	$\begin{array}{c} C_3H_7-n \\ \\ -N- \\ \\ OCH(CH_3)_2 \end{array}$
$\begin{array}{c} CH(CH_3)_2 \\ \\ -N- \\ \\ \text{C}_6\text{H}_4 \\ \\ CH_3 \end{array}$	$\begin{array}{c} CH_3 \\ \\ -N- \\ \\ \text{C}_6\text{H}_4 \\ \\ CH_3O \end{array}$
$\begin{array}{c} CH(CH_3)_2 \\ \\ -N- \\ \\ O-CH(CH_3)_2 \end{array}$	$\begin{array}{c} CH(CH_3)_2 \\ \\ -N- \\ \\ \text{C}_6\text{H}_4 \\ \\ CF_3 \end{array}$
$\begin{array}{c} CH_3 \\ \\ -N- \\ \\ \text{C}_6\text{H}_4 \\ \\ CH_3 \end{array}$	$\begin{array}{c} CH(CH_3)_2 \\ \\ -N- \\ \\ \text{C}_6\text{H}_4 \\ \\ Cl \\ \\ Cl \end{array}$

5

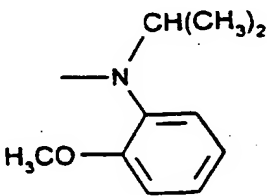
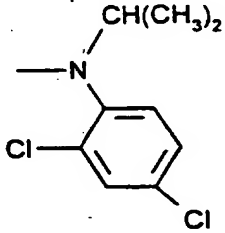
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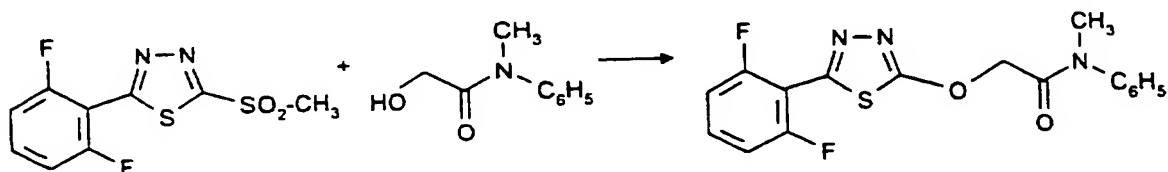
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Table 1

(Continued)

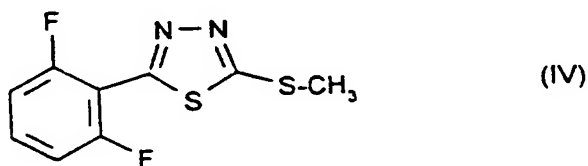
$\begin{array}{c} \text{R}^1 \\ \diagup \\ -\text{N} \\ \diagdown \\ \text{R}^2 \end{array}$	$\begin{array}{c} \text{R}^1 \\ \diagup \\ -\text{N} \\ \diagdown \\ \text{R}^2 \end{array}$
 <p>Chemical structure: A benzene ring with a methoxy group (H₃CO-) at the para position and a -N(CH₂CH₃)₂ group at the other para position.</p>	 <p>Chemical structure: A benzene ring with chlorine atoms (Cl-) at the 2 and 4 positions and a -N(CH₂CH₃)₂ group at the 1 position.</p>

If, for example, 2-(2,6-difluoro-phenyl)-5-methylsulphonyl-1,3,4-thiadiazole and N-methyl-hydroxyacetanilide are used as starting substances, the course of the reaction in the process according to the invention can be outlined by the following equation:



- 5 The compound 2-(2,6-difluoro-phenyl)-5-methylsulphonyl-1,3,4-thiadiazole of the formula (II) to be used as starting substance in the process according to the invention is still not known from the literature; it is, as a new substance, also a subject of the present application.

- 10 The new compound of the formula (II) is obtained when 2-(2,6-difluoro-phenyl)-5-methylthio-1,3,4-thiadiazole of the formula (IV)



is reacted with an oxidizing agent, such as e.g. hydrogen peroxide, if appropriate in the presence of a diluent, such as e.g. acetic acid and/or water, at temperatures between 0°C and 100°C (cf. the Preparation Examples).

- 15 The compound 2-(2,6-difluoro-phenyl)-5-methylthio-1,3,4-thiadiazole of the formula (IV) needed as a precursor is still not known from the literature; it is, as a new substance, likewise a subject of the present application.

The new compound of the formula (IV) is obtained when 2,6-difluoro-benzoic acid is

treated with phosphoryl chloride and then reacted with S-methyl dithiocarbamate (cf. the Preparation Examples).

Formula (III) provides a general definition of the hydroxyacetamides further to be used as starting substances in the process according to the invention for the preparation of the compounds of the general formula (I). In the formula (III), R^1 and R^2 preferably or in particular have those meanings which have already been indicated above as preferred or as particularly preferred for R^1 and R^2 in connection with the description of the compounds of the formula (I).

The hydroxyacetamides of the formula (III) are known and/or can be prepared by processes known per se (cf. US 4 509 971, US 4 645 525, US 4 334 073, DE 3 038 598, DE 3 038 636, EP 3 75 26, EP 34 87 37, DE 3 819 477).

Suitable diluents for carrying out the process according to the invention are the customary inert organic solvents. These include, in particular, aliphatic, alicyclic or aromatic, optionally halogenated hydrocarbons, such as, for example, pentane, hexane, heptane, petroleum ether, ligroin, benzine, benzene, toluene, xylene, chlorobenzene, dichlorobenzene, cyclohexane, methylcyclohexane, dichloromethane, chloroform, tetrachloromethane; ethers, such as diethyl ether, diisopropyl ether, t-butyl methyl ether, t-pentyl methyl ether, dioxane, tetrahydrofuran, ethylene glycol dimethyl ether or diethyl ether, diethylene glycol dimethyl ether or diethyl ether; ketones, such as acetone, butanone or methyl isobutyl ketone; nitriles, such as acetonitrile, propionitrile, butyronitrile or benzonitrile; amides, such as N,N-dimethylformamide, N,N-dimethylacetamide, N-methylformanilide, N-methyl-pyrrolidone or hexamethylphosphoramide; esters such as methyl acetate, ethyl acetate, n- or i-propyl acetate, n-, i- or s-butyl acetate; sulphoxides, such as dimethyl sulphoxide; alcohols, such as methanol, ethanol, n- or i-propanol, n-, i-, s- or t-butanol, ethylene glycol monomethyl ether or monoethyl ether, diethylene glycol monomethyl ether, diethylene glycol monoethyl ether; their mixtures with water or pure water.

The process according to the invention is preferably carried out in the presence of a suitable acid-binding agent. Those which are suitable are all customary inorganic or

organic bases. These include, for example, alkali metal or alkaline earth metal hydrides, hydroxides, amides, alkoxides, acetates, carbonates or hydrogen carbonates, such as, for example, lithium, sodium, potassium or calcium hydride, lithium, sodium or potassium amide, sodium or potassium methoxide, sodium or potassium ethoxide, sodium or potassium propoxide, aluminium isopropoxide, sodium or potassium tert-butoxide, sodium or potassium hydroxide, ammonium hydroxide, sodium, potassium or calcium acetate, ammonium acetate, sodium, potassium or calcium carbonate, ammonium carbonate, sodium or potassium hydrogen carbonate, and also basic organic nitrogen compounds, such as trimethylamine, triethylamine, tripropylamine, tributylamine, ethyldiisopropylamine, N,N-dimethylcyclohexylamine, dicyclohexylamine, ethyldicyclohexylamine, N,N-dimethylaniline, N,N-dimethyl-benzylamine, pyridine, 2-methyl-, 3-methyl- and 4-methyl-pyridine, 2,4-dimethyl-, 2,6-dimethyl-, 3,4-dimethyl- and 3,5-dimethyl-pyridine, 5-ethyl-2-methyl-pyridine, N-methylpiperidine, N,N-dimethylaminopyridine, diazabicyclooctane (DABCO), diazabicyclononene (DBN) or diazabicycloundecene (DBU).

The reaction temperatures can be varied within a relatively wide range when carrying out the process according to the invention. In general, it is carried out at temperatures between -50°C and +100°C, preferably at temperatures between -20°C and +60°C.

The process according to the invention is in general carried out under normal pressure. However, it is also possible to work at elevated or reduced pressure - in general between 0.1 bar and 10 bar.

To carry out the process according to the invention, the starting substances needed in each case are in general employed in approximately equimolar amounts. However, it is also possible to use one of the two components employed in each case in a relatively large excess. The reactions are in general carried out in a suitable diluent in the presence of an acid acceptor, and the reaction mixture is stirred for several hours at the temperature necessary in each case. Working up in the process according to the invention is in each case carried out according to customary methods (cf. the Preparation Examples).

The active compounds according to the invention can be used as defoliants, desiccants, agents for destroying broad-leaved plants and, especially, as weed-killers. By weeds, in the broadest sense, there are to be understood all plants which grow in locations where they are undesired. Whether the substances according to the invention act as total or selective herbicides depends essentially on the amount used.

The active compounds according to the invention can be used, for example, in connection with the following plants:

Dicotyledon weeds of the genera:

10 Sinapis, Lepidium, Galium, Stellaria, Matricaria, Anthemis, Galinsoga, Chenopodium, Urtica, Senecio, Amaranthus, Portulaca, Xanthium, Convolvulus, Ipomoea, Polygonum, Sesbania, Ambrosia, Cirsium, Carduus, Sonchus, Solanum, Rorippa, Rotala, Lindernia, Lamium, Veronica, Abutilon, Emex, Datura, Viola, Galeopsis, Papaver, Centaurea, Trifolium, Ranunculus and Taraxacum.

Dicotyledon cultures of the genera:

15 Gossypium, Glycine, Beta, Daucus, Phaseolus, Pisum, Solanum, Linum, Ipomoea, Vicia, Nicotiana, Lycopersicon, Arachis, Brassica, Lactuca, Cucumis and Cucurbita.

Monocotyledon weeds of the genera:

20 Echinochloa, Setaria, Panicum, Digitaria, Phleum, Poa, Festuca, Eleusine, Brachiaria, Lolium, Bromus, Avena, Cyperus, Sorghum, Agropyron, Cynodon, Monochoria, Fimbristylis, Sagittaria, Eleocharis, Scirpus, Paspalum, Ischaemum, Sphenoclea, Dactyloctenium, Agrostis, Alopecurus and Apera.

Monocotyledon cultures of the genera:

Oryza, Zea, Triticum, Hordeum, Avena, Secale, Sorghum, Panicum, Saccharum, Ananas, Asparagus and Allium.

25 However, the use of the active compounds according to the invention is in no way restricted to these genera, but also extends in the same manner to other plants.

The compounds are suitable, depending on the concentration, for the total combating of weeds, for example on industrial terrain and rail tracks, and on paths and squares with or without tree plantings. Equally, the compounds can be employed for combating weeds in perennial cultures, for example afforestations, decorative tree plantings, orchards, vineyards, citrus groves, nut orchards, banana plantations, coffee plantations, tea plantations, rubber plantations, oil palm plantations, cocoa plantations, soft fruit plantings and hopfields, in lawns, turf and pasture-land, and for the selective combating of weeds in annual cultures.

The compounds of the formula (I) according to the invention are particularly suitable for the selective combating of monocotyledon and dicotyledon weeds in monocotyledon and dicotyledon crops, especially pre-emergence.

The active compounds can be converted into the customary formulations, such as solutions, emulsions, wettable powders, suspensions, powders, dusting agents, pastes, soluble powders, granules, suspension-emulsion concentrates, natural and synthetic materials impregnated with active compound, and very fine capsules in polymeric substances.

These formulations are produced in a known manner, for example by mixing the active compounds with extenders, that is liquid solvents and/or solid carriers, optionally with the use of surface-active agents, that is emulsifying agents and/or dispersing agents and/or foam-forming agents.

In the case of the use of water as an extender, organic solvents can, for example, also be used as auxiliary solvents. As liquid solvents, there are suitable in the main: aromatics, such as xylene, toluene or alkyl-naphthalenes, chlorinated aromatics and chlorinated aliphatic hydrocarbons, such as chlorobenzenes, chloroethylenes or methylene chloride, aliphatic hydrocarbons, such as cyclohexane or paraffins, for example petroleum fractions, mineral and vegetable oils, alcohols, such as butanol or glycol as well as their ethers and esters, ketones, such as acetone, methyl ethyl ketone, methyl isobutyl ketone or cyclohexanone, strongly polar solvents, such as dimethylformamide and dimethyl sulphoxide, as well as water.

As solid carriers there are suitable:

for example ammonium salts and ground natural minerals, such as kaolins, clays, talc, chalk, quartz, attapulgit, montmorillonite or diatomaceous earth, and ground synthetic minerals, such as highly disperse silica, alumina and silicates, as solid carriers for
5 granules there are suitable: for example crushed and fractionated natural minerals such as calcite, marble, pumice, sepiolite and dolomite, as well as synthetic granules of inorganic and organic meals, and granules of organic material such as sawdust, coconut shells, maize cobs and tobacco stalks; as emulsifying and/or foam-forming agents there are suitable: for example non-ionic and anionic emulsifiers, such as polyoxyethylene
10 fatty acid esters, polyoxyethylene fatty alcohol ethers, for example alkylaryl polyglycol ethers, alkylsulphonates, alkylsulphates, arylsulphonates as well as albumen hydrolysis products; as dispersing agents there are suitable: for example lignin-sulphite waste liquors and methylcellulose.

Adhesives such as carboxymethylcellulose and natural and synthetic polymers in the
15 form of powders, granules or latexes, such as gum arabic, polyvinyl alcohol and polyvinyl acetate, as well as natural phospholipids, such as cephalins and lecithins, and synthetic phospholipids, can be used in the formulations. Further additives can be mineral and vegetable oils.

It is possible to use colorants such as inorganic pigments, for example iron oxide,
20 titanium oxide and Prussian Blue, and organic dyestuffs, such as alizarin dyestuffs, azo dyestuffs and metal phthalocyanine dyestuffs, and trace nutrients such as salts of iron, manganese, boron, copper, cobalt, molybdenum and zinc.

The formulations in general contain between 0.1 and 95 per cent by weight of active compound, preferably between 0.5 and 90%.

25 For controlling weeds, the active compounds according to the invention, as such or in the form of their formulations, can also be used as mixtures with known herbicides, finished formulations or tank mixes being possible.

Possible components for the mixtures are known herbicides, such as, for example, anilides, such as e.g. diflufenican and propanil; arylcarboxylic acids, such as e.g. dichloropicolinic acid, dicamba and picloram; aryloxyalkanoic acids, such as e.g. 2,4-D, 2,4-DB, 2,4-DP, fluroxypyr, MCPA, MCPP and triclopyr; aryloxy-phenoxy-alkanoic acid esters, such as e.g. diclofop-methyl, fenoxaprop-ethyl, fluazifop-butyl, haloxyfop-methyl and quizalofop-ethyl; azinones, such as e.g. chloridazon and norflurazon; carbamates, such as e.g. chlorpropham, desmedipham, phenmedipham and propham; chloroacetanilides, such as e.g. alachlor, acetochlor, butachlor, metazachlor, metolachlor, pretilachlor and propachlor; dinitroanilines, such as e.g. oryzalin, pendimethalin and trifluralin; diphenyl ethers, such as e.g. acifluorfen, bifenox, fluoroglycofen, fomesafen, halosafen, lactofen and oxyfluorfen; ureas, such as e.g. chlortoluron, diuron, fluometuron, isoproturon, linuron and methabenzthiazuron; hydroxylamines, such as e.g. alloxydim, clethodim, cycloxydim, sethoxydim and tralkoxydim; imidazolinones, such as e.g. imazethapyr, imazamethabenz, imazapyr and imazaquin; nitriles, such as e.g. bromoxynil, dichlobenil and ioxynil; oxyacetamides, such as e.g. mefenacet; sulphonylureas, such as e.g. amidosulfuron, bensulfuron-methyl, chlorimuron-ethyl, chlorsulfuron, cinosulfuron, metsulfuron-methyl, nicosulfuron, primisulfuron, pyrazosulfuron-ethyl, thifensulfuron-methyl, triasulfuron and tribenuron-methyl; thiocarbamates, such as e.g. butylate, cycloate, diallate, EPTC, esprocarb, molinate, prosulfocarb, thiobencarb and triallate; triazines, such as e.g. atrazine, cyanazine, simazine, simetryne, terbutryne and terbuthylazine; triazinones, such as e.g. hexazinone, metamitron and metribuzin; others, such as e.g. aminotriazole, benfuresate, bentazone, cinmethylin, clomazone, clopyralide, difenzoquat, dithiopyr, ethofumesate, fluorochloridone, glufosinate, glyphosate, isoxaben, pyridate, quinchlorac, quinmerac, sulphosate and tridiphane.

A mixture with other known active compounds, such as fungicides, insecticides, acaricides, nematocides, bird repellants, plant nutrients and agents which improve soil structure, is also possible.

The active compounds can be used as such, in the form of their formulations or in the use forms prepared therefrom by further dilution, such as ready-to-use solutions, suspensions, emulsions, powders, pastes and granules. They are used in the customary

manner, for example by watering, spraying, atomizing or scattering.

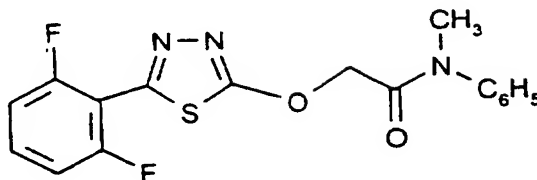
The active compounds according to the invention can be applied either before or after emergence of the plants. They can also be incorporated into the soil before sowing.

5 The amount of active compound used can vary within a substantial range. It depends essentially on the nature of the desired effect. In general, the amounts used are between 10 g and 10 kg of active compound per hectare of soil surface, preferably between 50 g and 5 kg per ha.

The preparation and use of the active compounds according to the invention can be seen from the following examples.

Preparation Examples:

Example 1



5 A solution of 0.4 g (10 mmol) of sodium hydroxide in 3 ml of water is added dropwise with stirring to a mixture of 2.76 g (10 mmol) of 2-(2,6-difluoro-phenyl)-5-methylsulphonyl-1,3,4-thiadiazole, 1.65 g (10 mmol) of N-methyl-hydroxyacetanilide and 35 ml of acetone, which is cooled to -15°C. The reaction mixture is then stirred at -15°C to 0°C for 12 hours. After acidifying with 2N hydrochloric acid, it is concentrated in a water-jet vacuum and the residue is stirred with a little water. The product which is obtained crystalline in this process is isolated by filtering off with suction.

3.15 g (87% of theory) of N-methyl-N-phenyl-2-[2-(2,6-difluorophenyl)-1,3,4-thiadiazol-5-yl-oxy]-acetamide of melting point 113°C are obtained.

15 The compounds of the formula (I) shown in Table 2 below can also be prepared, for example, analogously to Example 1 and corresponding to the general description of the preparation process according to the invention:

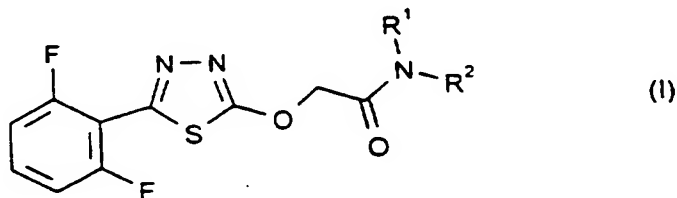
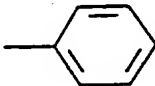
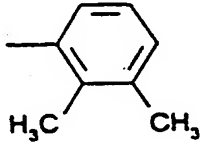


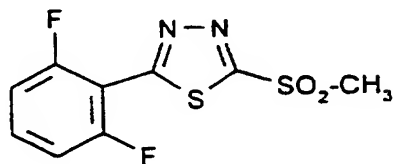


Table 2: Examples of the compounds of the formula (I)

Ex. No.	R ¹	R ²	Melting point (°C)
2	CH(CH ₃) ₂		107
5	3	C ₂ H ₅	70
	4	CH ₃	136
			
5	CH ₃		99
6		$\text{---}(\text{CH}_2)_4\text{---CH---}$ C ₂ H ₅	74
7		$\text{---}(\text{CH}_2)_4\text{---CH---}$ CH ₃	69
8	CH ₃	$\text{---CH---C}_2\text{H}_5$ CH ₃	61
10	9	C ₃ H ₇	70
	10	$\text{---}(\text{CH}_2)_6\text{---}$	91
11	$\text{---CH}_2\text{---CH=CH}_2$	$\text{---CH}_2\text{---CH=CH}_2$	59
12	CH(CH ₃) ₂	OCH(CH ₃) ₂	99
13	CH(CH ₃) ₂		128

Starting compound of the formula (II):

Example (II-1)

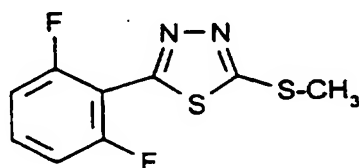


5 15 ml of a 35% strength aqueous hydrogen peroxide solution are added dropwise with stirring to a solution of 10.9 g (44 mmol) of 2-(2,6-difluoro-phenyl)-5-methylthio-1,3,4-thiadiazole in 80 ml of acetic acid, which is heated to 50°C to 60°C. The reaction mixture is stirred at 60°C for 60 minutes and then cooled. The product which is obtained crystalline after addition of ice-water is isolated by filtering off with suction.

10 10.6 g (87% of theory) of 2-(2,6-difluoro-phenyl)-5-methylsulphonyl-1,3,4-thiadiazole of melting point 133°C are obtained.

Starting compound of the formula (IV):

Example (IV-1)



15 45.3 g (0.37 mol) of S-methyl dithiocarbazate are added in portions at about 10°C with stirring to a mixture of 58.6 g (0.37 mol) of 2,6-difluoro-benzoic acid and 300 ml of phosphoryl chloride (POCl₃) and the reaction mixture is then additionally stirred at about 20°C for about 60 minutes. It is then concentrated in a water-jet vacuum, and the residue is taken up in chloroform and washed with ice-water and then with saturated sodium hydrogen carbonate solution. After drying the organic phase with sodium sulphate, it is filtered and the filtrate is concentrated in a water-jet vacuum. The

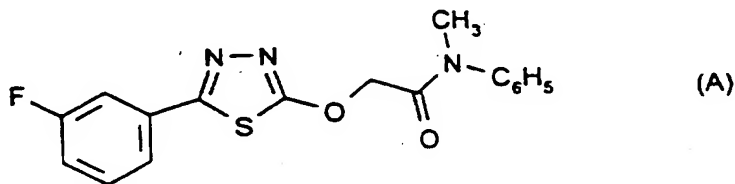
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residue is digested with isopropanol and the product which is obtained crystalline is isolated by filtering off with suction.

86 g (95% of theory) of 2-(2,6-difluoro-phenyl)-5-methylthio-1,3,4-thiadiazole of melting point 45°C are obtained.

5 Use Examples:

The compound mentioned below is used as a comparison substance in the use examples.



- 10 N-Methyl-N-phenyl-2-[2-(3-fluoro-phenyl)-1,3,4-thiadiazol-5-yl-oxy]-acetamide
(disclosed in EP-A 53 75 43).

Example A

Pre-emergence test

Solvent: 5 parts by weight of acetone

Emulsifier: 1 part by weight of alkylaryl polyglycol ether

- 5 To produce a suitable preparation of active compound, 1 part by weight of active compound is mixed with the stated amount of solvent, the stated amount of emulsifier is added and the concentrate is diluted with water to the desired concentration.

- 10 Seeds of the test plants are sown in normal soil. After 24 hours, the soil is watered with the preparation of the active compound. It is expedient to keep constant the amount of water per unit area. The concentration of the active compound in the preparation is of no importance, only the amount of active compound applied per unit area being decisive. After three weeks, the degree of damage to the plants is rated in % damage in comparison to the development of the untreated control.

The figures denote:

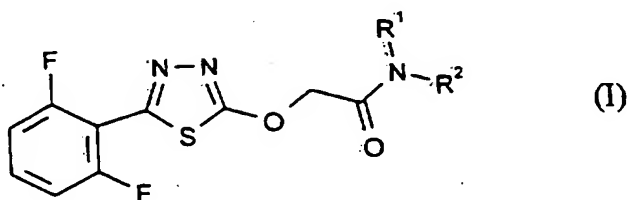
- 15 0% = no action (like untreated control)
100% = total destruction

- In this test, for example, the active compounds according to Preparation Examples 6, 8 and 10 show, combined with very good tolerability to crop plants, such as e.g. barley, considerably stronger action against weeds than the known compound A.
- 20 Likewise, the active compounds according to Preparation Examples 1, 7, 9, 11, 12 and 13 exhibit strong action against various weeds, combined with good tolerability to crop plants, such as e.g. wheat and soya beans.

- 25 It will be understood that the specification and examples are illustrative but not limitative of the present invention and that other embodiments within the spirit and scope of the invention will suggest themselves to those skilled in the art.

Patent Claims

1. 2-[2-(2,6-Difluoro-phenyl)-1,3,4-thiadiazol-5-yl-oxy]-acetamides of the general formula (I)



5 in which

R¹ represents alkyl, alkenyl, alkynyl or arylalkyl, each of which is optionally substituted, and

R² represents alkyl, alkenyl, alkynyl, cycloalkyl, cycloalkenyl, alkoxy, alkenyloxy, alkynyloxy, arylalkyl or aryl, each of which is optionally substituted, or

10

R¹ and R², together with the nitrogen atom to which they are bonded, represent an optionally substituted, monocyclic or bicyclic heterocycle.

2. Compounds of the formula (I) according to Claim 1, characterized in that therein

15

R¹ represents C₁-C₈-alkyl (which is optionally substituted by fluorine, chlorine, cyano or C₁-C₄-alkoxy), C₂-C₈-alkenyl (which is optionally substituted by fluorine and/or chlorine), C₂-C₈-alkynyl or benzyl (which is optionally substituted by fluorine, chlorine, cyano, C₁-C₄-alkyl or C₁-C₄-alkoxy), and

20

R² represents C₁-C₈-alkyl (which is optionally substituted by fluorine, chlorine, cyano or C₁-C₄-alkoxy), C₂-C₈-alkenyl (which is optionally

5

substituted by fluorine and/or chlorine), C₂-C₈-alkynyl, C₃-C₆-cycloalkyl (which is optionally substituted by chlorine and/or C₁-C₃-alkyl), cyclopentenyl or cyclohexenyl, C₁-C₈-alkoxy (which is optionally substituted by methoxy or ethoxy), C₃-C₄-alkenyloxy, benzyl (which is optionally substituted by fluorine, chlorine, cyano, C₁-C₄-alkyl or C₁-C₄-alkoxy) or phenyl (which is optionally substituted by fluorine, chlorine, bromine, cyano, nitro, C₁-C₄-alkyl, trifluoromethyl, C₁-C₄-alkoxy or C₁-C₄-alkylthio), or

10

R¹ and R², together with the nitrogen atom to which they are bonded, represent a monocyclic or bicyclic, five- to seven-membered nitrogen heterocycle which is optionally mono- to trisubstituted by C₁-C₃-alkyl.

3. Compounds of the formula (I) according to Claim 1, characterized in that therein

15

R¹ represents methyl, ethyl, n- or i-propyl, n-, i-, s- or t-butyl, n-, i-, s- or t-pentyl (which in each case are optionally substituted by fluorine, chlorine, cyano, methoxy or ethoxy), propenyl, butenyl, pentenyl, propinyl, butinyl, pentinyl or benzyl,

20

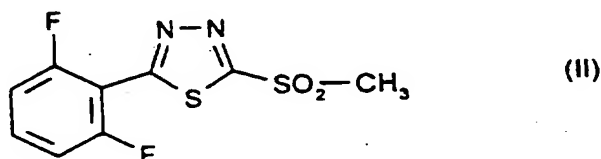
R² represents methyl, ethyl, n- or i-propyl, n-, i- or s-butyl, n-, i- or s-pentyl, n-, i- or s-hexyl (which in each case are optionally substituted by fluorine, chlorine, cyano, methoxy or ethoxy), propenyl, butenyl, pentenyl, propinyl, butinyl or pentinyl, cyclopentyl or cyclohexyl (which in each case are optionally substituted by methyl and/or ethyl), cyclohexenyl, methoxy, ethoxy, n- or i-propoxy, n-, i- or s-butoxy, n-, i- or s-pentyloxy (which in each case are optionally substituted by methoxy or ethoxy), benzyl (which is optionally substituted by fluorine, chlorine and/or methyl) or phenyl (which is optionally substituted by fluorine, chlorine, bromine, cyano, nitro, methyl, ethyl, trifluoromethyl, methoxy or ethoxy), or

25

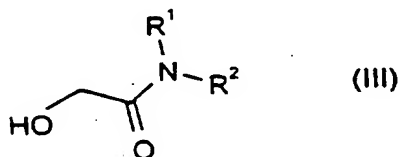
R¹ and R², together with the nitrogen atom to which they are bonded, represent

piperidinyl which is optionally mono- to trisubstituted by methyl and/or ethyl, pyrrolidinyl which is optionally mono- or disubstituted by methyl and/or ethyl, perhydroazepinyl or 1,2,3,4-tetrahydro(iso)quinolinyl.

4. Process for the preparation of compounds of the formula (I) according to Claim 1, characterized in that 2-(2,6-difluoro-phenyl)-5-methylsulphonyl-1,3,4-thiadiazole of the formula (II)



is reacted with hydroxyacetamides of the general formula (III)



10 in which

R¹ and R² have the meaning indicated in Claim 1,

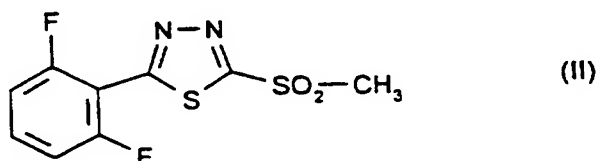
if appropriate in the presence of a diluent and if appropriate in the presence of an acid-binding agent.

5. Herbicidal compositions, characterized in that they contain at least one compound of the formula (I) according to Claim 1.
6. Use of compounds of the general formula (I) according to Claim 1 for combating undesired plant growth.
7. Method of combating weeds, characterized in that compounds of the general

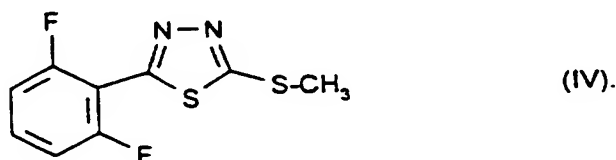
formula (I) according to Claim 1 are allowed to act on the weeds or their habitat.

8. Process for the production of herbicidal compositions, characterized in that compounds of the general formula (I) according to Claim 1 are mixed with extenders and/or surface-active agents.

9. 2-(2,6-Difluoro-phenyl)-5-methylsulphonyl-1,3,4-thiadiazole of the formula (II) and 2-(2,6-difluoro-phenyl)-5-methylthio-1,3,4-thiadiazole of the formula (IV)



and



10. A compound of the formula (I) according to Claim 1, as hereinbefore specifically identified.
11. Process according to Claim 4, when carried out substantially as hereinbefore described with reference to any one of Examples 1 to 13.
12. A compound of the formula (I) according to Claim 1, whenever produced by the process according to claim 4 or 11.
13. Herbicidal composition containing at least one compound according to Claim 10 or 12.

14. Use of a compound according to Claim 10 or 12 for combating undesired plant growth.
15. Method of combating weeds, characterised in that a compound according to Claim 10 or 12 is allowed to act on the weeds or their habitat.
16. Process for the production of a herbicidal composition, characterised in that a compound according to Claim 10 or 12 is mixed with one or more extenders and/or surface-active agents.

Patents Act 1977
Examiner's report to the Comptroller under Section 17
(The Search report)

Application number
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Relevant Technical Fields

- (i) UK Cl (Ed.O) C2C
 (ii) Int Cl (Ed.6) C07D

Search Examiner
 MR S J QUICK

Date of completion of Search
 18 JANUARY 1996

Databases (see below)

(i) UK Patent Office collections of GB, EP, WO and US patent specifications.

Documents considered relevant following a search in respect of Claims :-
 1-16

(ii) ONLINE: CAS ONLINE

Categories of documents

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Category	Identity of document and relevant passages	Relevant to claim(s)
X	EP 0537543 A1 (BAYER) see especially Examples 25-27, 29-33 and 58-61 and page 2, lines 1-2 and 30ff (acknowledged in this application)	1-16

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